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Investigating Nitriding Parameters for AISI H13 Tool Steel: A Microhardness Study Using Full Factorial Design

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Abstract: This research investigates the parameters affecting gas nitriding to increase the surface microhardness of AISI H13 tool steel. Used full factorial experimental design testing combinations of nitriding temperature (200°C, 300°C, 400°C) with nitriding time (12, 24, 36 hours) under constant ammonia flow (0.5 L/min, atmospheric pressure conditions. The Vickers microhardness ($HV_{0.5}$) test results demonstrated that hardness reaches its maximum at 400°C among temperature treatment conditions. The hardness peak value reached 1062 HV during the optimal condition at 400°C for 24 hours but embrittlement caused the hardness to decrease by 4% when the process exceeded 36 hours. Hardness reached its peak value of 952 HV during 24 hours of treatment at 300°C before reduction happened, although 200°C produced only small enhancements between 853 HV and 867 HV because of inadequate nitrogen migration. Data analysis through interaction and contour plots demonstrated temperature as the determining factor for experimental results after 24-hour processing time.

Introduction

The applications of AISI H13 tool steel in die-casting, extrusion, and hot forging depend on its high-temperature strength combined with thermal fatigue resistance and toughness characteristics (Li et al., 2016; Amirabdollahian et al., 2021; Walczak et al., 2022; Maly et al., 2024). Tool failures emerge early because cyclic thermo-mechanical loading causes surface damage, yet industry requires advanced surface engineering solutions (Ahmad et al., 2020). Gas nitriding achieves exceptional results for tool steel surface enhancement because it improves wear resistance together with fatigue lifetime without affecting material properties inside the bulk material (Staia et al., 2009; Menthe et al., 2000; Le et al., 2021; Al-Saraireh and Suhymat, 2024). The sub-critical temperature range of 450–600°C allows thermochemical nitrogen to diffuse into steel-producing hard iron nitride compounds (Fe_4N and $Fe_{2-3}N$), which boost surface characteristics of corrosion resistance and durability (Hacısalihoğlu et al., 2021). Previous harsh and general assessments between nitriding parameters and material hardness have not satisfied the need within commercial settings for precise optimization between cost-effectiveness and practical results (Parlinska-Wojtan et al., 2006). The combination of extended nitriding durations causes two problems: elevated energy costs as well as material embrittlement due to excessive nitriding (Kulkarni et al., 2007; Bohinc et al., 2020; Patel and Gandhi, 2024), although brief treatments deliver few gains. A systematic study evaluates gas nitriding parameters for AISI H13 steel under three temperature conditions (200°C, 300°C, 400°C) and solution times (12, 24, 36 hours) for the purpose of:

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#A combination of temperature and nitriding time must be determined to achieve the highest possible surface microhardness values.

#The study evaluated decreased performance benefits that resulted from longer treatment periods at different temperatures.

#Establish practical guidelines for industrial tooling applications

Materials and Methods

Material

AISI H13 tool steel samples measuring 20 mm × 20 mm × 5 mm originated from commercial material. Industrial surface engineering studies needed materials with high hot hardness and thermal fatigue resistance, so the researchers selected this material (Sousa et al., 2021).

Figure 1. Nirtiding Sample.

The material composition satisfied ASTM A681 requirements through its content of 0.40% C and 5.20% Cr together with 1.40% Mo and 1.00% V weight percent. The standard metallographic technique was applied to all test specimens before nitriding to ensure their surfaces were equally prepared (Mydłowska et al., 2017).

Nitriding Process

Ammonia (NH_3) gas nitriding process was carried out in a laboratory-scale heat treatment furnace which has temperature and ammonia gas flow control systems. The full factorial design comprising two independent variables was nitriding temperature (200°C, 300°C and 400°C) and nitriding time (12 hours, 24 hours, 36 hours). In the experimental research, there were indeed nine factorial combinations of conditions as presented in Table 1 below. Specifications of temperature and time have been investigated only; all the other factors influencing the process have been kept constant (Mendoza and Ibarra, 2023). The constants which remained constant during the course of the experimentation are as follows:

#A consistent nitrogen supply during diffusion is achieved through using Ammonia Gas Flow at 0.5 L/min.

#The furnace operates at 1 atm (standard atmospheric pressure) to prevent changes resulting from changes in pressure.

#A heating process at 5°C per minute is applied uniformly to achieve the target temperature.

#Slow cooling happens inside the furnace atmosphere of ammonia to maintain the nitride layer structure.

Table 1. Experimental Nitriding Conditions.

Temperature (°C)	Nitriding Time (hours)
200	12
200	24
200	36
300	12
300	24
300	36
400	12
400	24
400	36

Results and Discussion

Microhardness Measurement

The Vickers hardness tester evaluated surface microhardness of the samples through a calibrated testing system, which required a 500 g ($HV_{0.5}$) load for 10–15 seconds at five locations on each sample surface. Surface measurements under 500 g ($HV_{0.5}$) pressure were performed during a 10–15-second testing period across five randomly chosen spots on the nitrided specimen surface to address possible surface unevenness. Research findings were based on the mean values from five microhardness measurements, which served as standard microhardness values (HV) for experimental protocols. The hardness measurement procedure produces dependable data which meets the specifications mentioned in ASTM E384 standards for micro-indentation testing. The observed data records average microhardness values, which are presented in Table 2 for additional analysis.

Main Effects and Interaction Plots

A higher S/N ratio with better characteristics was used and was calculated with the help of the Minitab 19 software for the experimental tests. The values of the complete S/N ratio are S/N ratio for the first test. From Graph 4.4, it is observed that the optimum Wear Rate was in the highest values of the response graph. The optimal input parameters were Reinforcement 2% (level

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1), Load 25 N (level 1) and Temp 80ºC (level 3). The graph shows the effect of the control factors on Brass material. The configuration of the operating parameters with the highest ratio always provides the optimum quality with a minimum variation. The graph shows the relationship change when the control factor configuration was changed from one level to another.

improvements between 12 and 24 hours at 200°C and 300°C testing temperatures, but it diminishes during 36 hours of testing despite the increase in exposure nitriding time to the temperature. The maximum hardness point of 1062 HV occurs at 24 hours at 400 degrees Celsius with an absence of further hardness enhancement in the 36-hour sample phase.

Table 2. Observed Microhardness Data.

Temperature (°C)	Nitriding time (hours)	Microhardness (HV)
200	12	858
200	24	867
200	36	853
300	12	918
300	24	952
300	36	892
400	12	1003
400	24	1062
400	36	1020

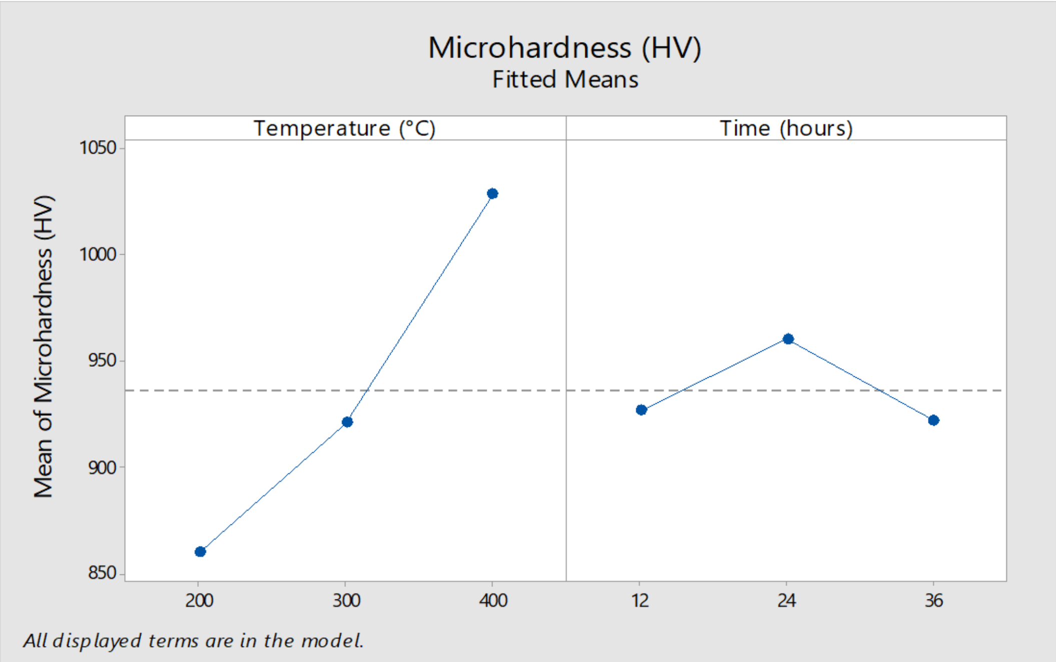

Figure 2. Main Effects Plot for Microhardness.

Temperature Effect: The microhardness values rise smoothly as temperature increases during the experiment. At 200°C, the fitted means range around 850–870 HV, with a slight increase from 12 to 24 hours and a dip at 36 hours. The value of microhardness increases to 900–950 HV and reaches its peak during 24 hours at 300°C. The test specimen experienced peak microhardness of 1000–1060 HV at 400°C, achieving both maximum values at 24 hours (1062 HV) and dropping to 1020 HV at 36 hours.

Nitriding time Effect: The quantity of time impacts microhardness measurements differently depending on the temperature value. The microhardness shows

The observed that optimized condition exists at 400°C for 24 hours since it reaches a maximum hardness level of approximately 1060 HV.

The results show that temperature-time combinations significantly influence the nitrided AISI H13 tool steel's microhardness levels due to the interaction plot's split lines. During the first 12 hours at temperatures between 200°C and 300°C and 400°C, the microhardness measurements remain approximately 850 HV to 1000 HV. The lines display distinct separating patterns when nitriding time reaches 24 hours because 400°C establishes the highest microhardness level of 1062 HV, showing enhanced diffusion capability during this

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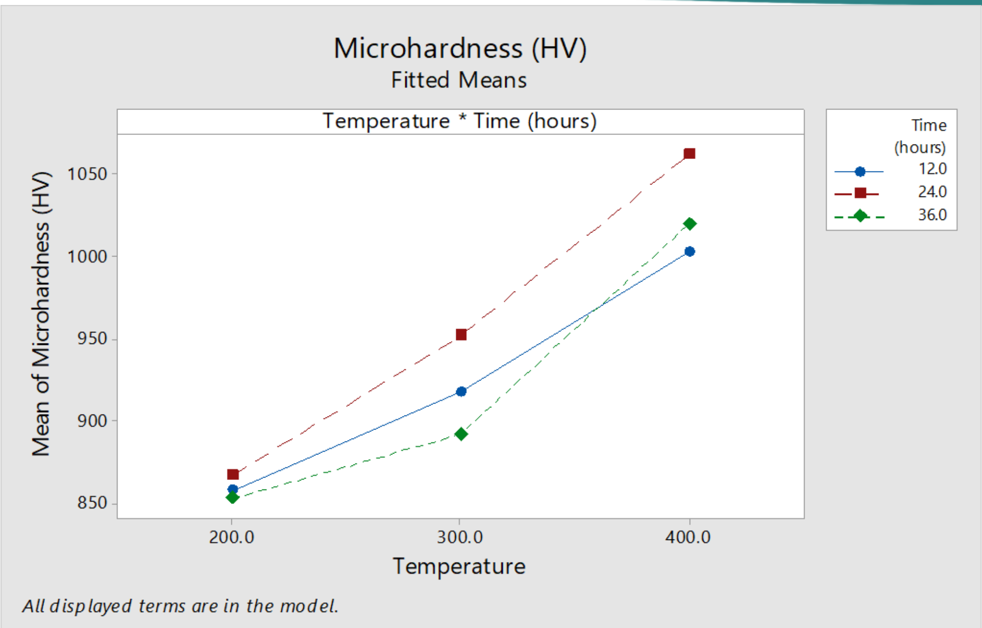

Figure 3. Interaction Plot for Microhardness.

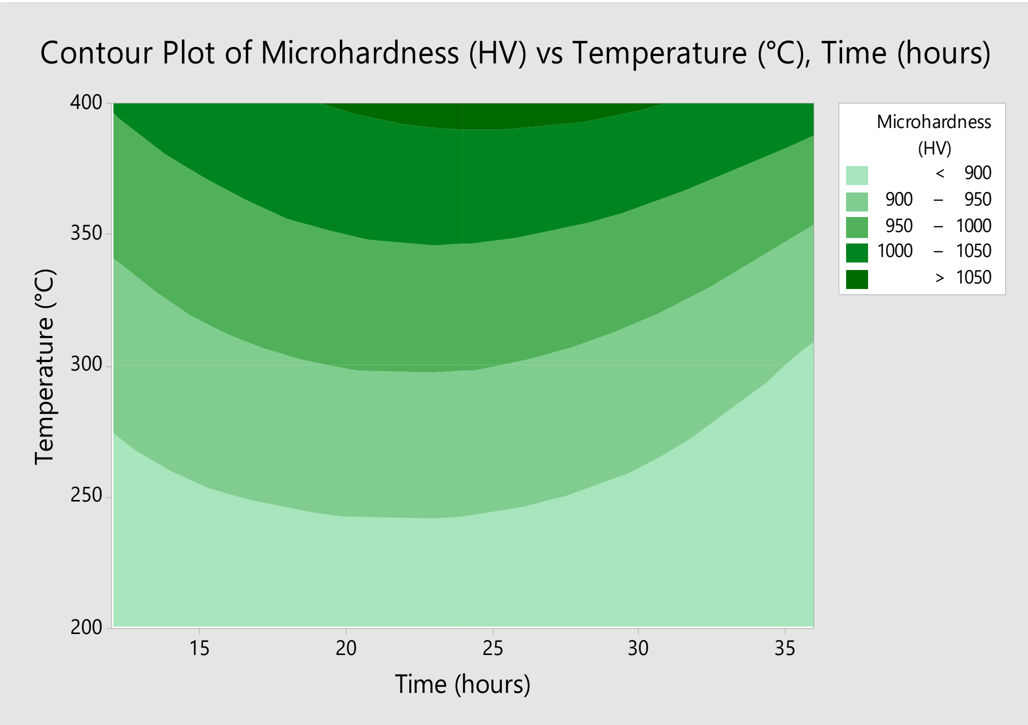

Figure 4. Interaction Plot for Microhardness.

period. The values show minor convergence between these time points, where 400°C drops to 1020 HV, but 300°C reaches 892 HV, while 200°C ends at 853 HV, which indicates both stabilizing and reducing hardness gain potential. The microhardness trends at different temperatures demonstrate a specific pattern because the measurements at 400°C show a rapid rise from 12 to 24 hours, which reaches its peak at 24 hours before dropping slightly during 36 hours, indicating an ideal diffusion timeframe. During the 24-hour period, the 300°C line reached 952 HV before beginning its decline at 36 hours, yet the 200°C line displayed steady HV between 853-867. Temperature controls nitrogen diffusion rates according to these results. These non-parallel lines demonstrate that microhardness measurement results significantly vary according to temperature levels, so the tested period of 24 hours at 400°C proves to be optimal. The measurements taken at 36 hours, regardless of temperature, indicate a possible limitation affecting nitrogen diffusion while showing signs of harmful compound layer formation that threatens surface quality.

Contour plot

Your figure's visual representation demonstrates how the AISI H13 tool steel microhardness level (HV) changes at different temperature (°C) and time (hours)

intervals throughout the gas nitriding treatment.

Temperature and nitriding time together affect the surface microhardness of AISI H13 tool steel according to the contour plot. Temperature plays a stronger role in hardening development than time exposure because the hardness bands display horizontal orientation. The surface hardness stays below 900 HV during nitriding at 200°C and at all time intervals because nitrogen diffusion is hindered at these subcritical temperatures. Surface material hardness undergoes substantial improvement from 900 to 1000 HV when the temperature reaches 300°C at the 24-hour mark. The longevity of 36 hours in the nitriding process shows no additional improvements in hardness properties, which might be caused by both treatment saturation and short-lived positive impacts from lengthening the duration. The highest microhardness increase occurs at 400°C since the material reaches over 1050 HV after 24 hours of treatment. The combination of optimal nitrogen diffusion and compound layer formation happens at this particular treatment condition. At 36 hours of exposure, hardness shows a decrease until it reaches the 1000–1050 HV range, which may stem from over-nitriding processes or unstable nitride layer formation and grain coarsening effects. Temperature control during nitriding processes demonstrates higher significance than extended exposure duration for achieving top surface properties, especially when operating at 400°C. The combination of 400°C temperature treatment for 24 hours represents the best condition because it achieves maximum microhardness effectiveness while avoiding negative effects of prolonged exposure.

Response Optimization Analysis

The objective of the response optimization method is to achieve the conception of the microhardness (HV) for nitrided AISI H13 tool steel from the gas nitriding process. The set values which control the optimization symbols are given below:

Table 3. Optimization Solution.

Response	Goal	Variable	Setting
Microhardness (HV)	Maximum	Temperature (°C)	400
		Nitriding Time (hours)	24

Conclusions and Future Directions

Conclusions

The optimal gas nitriding parameters for AISI H13 tool steel occur at 400°C for 24 hours, which generates a maximum microhardness of 1062 HV. The 36-hour nitriding process causes surface hardness to reach 1020 HV and thus represents a 4% decrease from the peak hardness of 1062 HV at 24 hours because prolonged nitriding has negative consequences such as embrittlement. The combination of a treatment temperature of 300°C with a duration of 24 hours produces the highest resulting microhardness value of 952 HV yet this value decreases to 892 HV when the time extension reaches 36 hours. At 200°C, the microhardness shows minimal movement, ranging between 853 HV and 867 HV, indicating lower temperature effectiveness for diffusion processes.

Research shows that accurate temperature maintenance at 400°C generates better microhardness results than what is achieved by keeping the material in the furnace excessively long. This leads to an efficient and affordable solution.

The findings offer concrete implementation recommendations for industrial tooling industries which depend on surface durability enhancement and validate the optimal nitriding condition at 400°C for 24 hours.

Future Directions

Further research for the duplex surface treatment process and the tribo-mechanical characteristics of the coated AISI H13 tool steel are as follows: Therefore, in light of the conclusions made in this study, the following areas should be examined to better understand and develop these coatings:

Advanced Multilayer and Nanocomposite Coatings: This indicates that the following studies could be directed towards the use of superior multilayer or nanocomposite coatings like TiAlN/Crn, Tin/AlCrN and TiSiN with increased thermal stability, hardness and wear resistance. With such applications of the power metallurgical process, these coatings can be designed to meet various services that have high contact loads and thermal stresses.

High-Temperature Tribological Testing: Wear testing performed at such temperatures (300–600°C) can more accurately replicate the situation typical for real service conditions. This would show cycle decay, oxidation capabilities and coating bond endurance, particularly the hot working tool steel used in manufacturing.

Optimizing the Wear Resistance using Taguchi Method. It can also be generalized that in the current work with the Taguchi approach, several additional factors could be further taken into consideration, such as the sliding speed, counter material type, and humidity levels. If it is possible to optimize more than one response variable, a Taguchi–GRA or a Taguchi ANOVA can be used to develop a multi-response optimization technique.

Conflict of interest

None

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